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(54) Composition and process for gelatin-free soft capsules

Zusammensetzung und Verfahren zur Herstellung von weichen Kapseln ohne Gelatin

Composition et procédé pour des capsules molles sans gélatine

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Description

BACKGROUND OF THE INVENTION

[0001] The present invention relates to a polymer composition comprised of gellan, carrageenan and mannan gums and a process for producing flexible films for encapsulation comprising the gellan, carrageenan and mannan gum composition. Advantageously, soft capsules of the above composition are produced using the novel process.

[0002] The art reveals that processes and compositions for encapsulation or producing soft capsules generally are composed of gelatin or animal based gels. Soft capsules have primarily been made from low bloom gelatin which melts at low temperatures so that in the manufacturing process two sheets of gelatin are sealed simultaneously with the filling of the capsule. There is a need, however, for producing soft capsules containing liquid or solid contents selected from food or nutritional supplements including vitamins or minerals and the like wherein the capsules are edible and derived from non animal based sources and the capsules can be produced on the existing machinery which is utilized to produce the gelatin based capsules and films. Various polysaccharides and various processes for making them and using them are known. See: U.S. 4,326,052; 4,326,053; 4,377,636; 4,385,123; 4,647,470; 4,746,528 and 5,112,445. U.S. Pat. No. 4,746,528 describes combinations of gellan, xanthan gum and a galactomannan and/or glucomannan gum used to produce elastic gels. U.S. Pat. No. 4,647,470 describes blends of low-acetyl gellan gum with xanthan gum and locust bean gum, konjak, tara or cassia gums which are useful for modifying the brittleness of gellan food products. Gelled food product compositions comprising blends of gellan gum/K-carrageenan gum and mannan gums in weight ratios of about 80:20 to 10:90 of K-carrageenan to mannan and about 95:5 to 20:80 of gellan gum to (K-carrageenan/mannan) are known. See GB 2219803 A.

[0003] The art also reveals that heretofore many of the film-forming water soluble materials have high melting temperatures and/or decompose at melting temperatures thus rendering them unsuitable for producing non-animal based capsules. There is an additional need to provide soft capsules which may be utilized to encapsulate bath oils and the like wherein the capsular material is composed of non-gelatin based sources and is water soluble. There is an additional need for novel processes related to the production of non-animal based soft capsules or flexible films such as those claimed and disclosed in the present invention. The present invention therefore satisfies the need for producing capsules wherein the particular non-animal derived compositions comprise suitable material which has the essential low melting and stability characteristics of the animal derived gelatins and thus can be produced on existing capsule machinery.

SUMMARY OF THE INVENTION

[0004] There is a need for non-animal sourced, i.e. gelatin-free, encapsulating polymer systems that will remelt under controlled conditions to form soft capsules to seal the encapsulated contents that may be selected from liquid or solid active or inactive ingredients within two polymer sheets.

[0005] The present invention therefore involves a trigum blend comprising (a) low-acyl gellan gum with a relative weight percentage of about 20%; (b) k-carrageenan gum with a relative weight percentage of about 50%; (c) locust bean gum or konjak gum or mixtures of locust bean gums and konjak gums in a ratio of 1 : 100 to 100 : 1 with a relative weight percentage of about 25%; and potassium citrate in a relative weight percentage of about 9%. The invention also concerns the use the inventive trigum blend in a gelatin-free soft capsule, the trigum comprising from 1 to 10 weight percent of the total weight of the capsule. In a further aspect the invention pertains to a process for producing a gelatin-free saft capsule wherein the capsule contents are solid or liquid and contain the trigum blend of the invention.

[0006] In order to achieve remelting of the film composition at a soft capsule processing temperature of less than 100 °C, sufficient water must be available to inhibit polymer association and subsequent increase in the melting temperature above 100°C.

DETAILED DESCRIPTION OF THE INVENTION

[0007] The present invention concerns a trigum blend comprising a low-acyl gellan gum with a relative weight percentage of about 20% k-carrageenan gum with a relative weight percentage of about 50% and locust bean gum or konjak gum or mixtures of locust bean gums and konjak gums in a ratio of 1 : 100 to 100 ; 1 with a relative weight percentage of about 25%; and potassium citrate in a relative weght percentage of about 9%.

[0008] The tri-gum blend concentration of the film-forming composition ranges from 1 to 10 weight percent of the total weight of the capsule. In order to achieve remelting of the film composition at a soft capsule processing temperature of less than 100 °C, sufficient water must be available to inhibit polymer association and subsequent increase in the melting temperature above 100°C.

[0009] The melting property of the flexible film disclosed in the present application is critical to the proper sealing of

the capsules produced during production of said soft capsules. The present invention makes use of a unique combination of hydrocolloids which interact to give synergistic film properties. Furthermore, by controlling the solids content of the film during the encapsulation process, a melting temperature of less than 100° C for the polymer composition within the scope of the present invention is achieved. The compositions and processes of the present invention have numerous advantages including biodegradability, strength, thermal reversibility, water solubility and reduced processing time.

[0010] Gellan gum refers to the extracellular polysaccharide obtained by the aerobic fermentation of the microorganism, Pseudomonas elodea, in a suitable nutrient medium. As used hereafter, "gellan gum" shall refer to low acetyl (LA) gellan gum which has an acetyl level of 0.3 to 0 % weight.

[0011] Locust bean gum (lbg) is an extract of the locust bean or carob, Ceratonia siligua and is a member of the galactomannan class. It is commercially available and used as a stabilizer in various food products. Konjac or konjac gum is a glucomannan extracted from the plant Amorphophallus konjac.

[0012] k-carrageenan is a hydrocolloid obtained by extraction with water or other polar solvents from some members of the algae class Rhodophyceae (red algae) and consists of a mixture of the ammonium, calcium, magnesium, potassium, and sodium esters of galactose and 3-6-anhydrogalactose copolymers. Carrageenans include the κ, λ, and τ forms. These additives are used predominantly as suspending or gelling agents in the pharmaceutical or food industries.

[0013] The present invention is directed to a tri-combination of the above components in particular ratios which under the appropriate conditions as described herein may be utilized to produce polymeric films useful in producing non-gelatin capsules which are soft and water soluble. The tri-combination may be formulated and is preferrably formulated in the following ratios for the film-forming polymeric compositions:

gellan gum	k-carrageenan	locust bean gum
0.100	0.225	0.675
0.100	0.675	0.225
0.100	2.475	0.7425
0.800	1.900	0.950
5.000	1.250	3.750
5.000	3.750	1.250

[0014] This tri-gum combination or a combination wherein a konjak gum is substituted for the locust bean gum or any tri-combination within the scope of the present invention may be further combined with a salt such as potassium citrate and additional ingredients such as sorbitol, glycerine, corn syrup and deionized water to form a film-forming polymeric composition. Sodium citrate and potassium chloride may also be added to forn an aqueous composition useful in the preparation of film-forming compositions. Sesquestrants selected from a sodium or potassium phosphate or citrate salt or combinations thereof may be utilized in the present invention. For example, sodium phosphate and/or sodium citrate may be added to the composition and are useful in chelating the divalent ions such as magnesium and calcium and allow complete hydration of the gellan gum. The added potassium salt level in the gum/film forming composition is useful as it relates to the melting and setting temperature of the film.

[0015] The following gelling salts may be utilized in the present invention: sodium chloride, sodium sulfate, and other sodium salts of appropriate orgainic or inorganic acids. Potassium sulfate and other potassium salts of appropriate organic or inorganic acids may also be utilized as gelling salts in the present invention. One skilled in the art will appreciate that under certain circumstances and conditions, certain ions are necessary to gel certain gums such as the kappa form of a carrageenan gum. Therefore, when κ-carrageenan is used, potassium ions must be present to obtain the maximum performance of the gum. Furthermore, the use of mono- or di-valent ions to gel gellan gum is determined by the requirement of gel texture and modulus.

[0016] A process for preparing compositions within the scope of the present invention and for manufacturing soft capsules from the compositions comprises the steps of:

(1) A tri-gum blend comprising gellan gum, locust bean gum and k-carrageenan gum in the relative ratios as described above such as, for example, (a) low-acyl gellan gum with a relative weight percentage of about 20%; (b) k-carrageenan gum with a relative weight percentage of about 50%; (c) locust bean gum with a relative weight percentage of about 25%; and a salt such as potassium citrate in a relative weight percentage of about 9% wherein the potassium citrate is used as a sequestrant/gelling salt is mixed into cold (20-30°C) deionized water and then the ingredients sorbitol, glycerine and corn syrup are added with agitation. The plasticizers or other reagents useful in the present invention may be selected from sorbitol, glycerine, propylene glycol, polyethylene glycol, corn syrup,

sucrose, fructose or combinations thereof. The mixture is then heated to a temperature of about 75-100 °C. Advantageously, the mixture is heated to 90°C with agitation and held at this temperature for about 10 minutes;

(2) The hot solution produced in step 1 is then, for example, transferred to an encapsulation machine wherein a 30-40 mil film is formed on rotating steel drums. Two films thus formed on the steel drums proceed through rotating dies that are designed to simultaneously form, cut and fill various sized and shaped capsules. The fill material may be either solid or liquid material. The edges of the capsule formed in this process are heat and pressured sealed and the filled capsules are then washed, dried to a pre-determined moisture content such as 3-4% and packaged. The moisture content of the film during the encapsulation process controls the melting temperature and the proper sealing of the capsules.

[0017] In the above process, the locust bean gum can readily be substituted with a mannan gum such as konjak gum or mixtures of locust bean gums and konjak gums in a ratio of 1:100 to 100:1. Gelatin free soft capsules utilizing these compositions may readily be prepared.

EXAMPLES

[0018] The following gum-blends and/or gum blend aqueous compositions may readily be prepared. The relative ratios of the gums and additional reagents are generally expressed in relative weight percentages. The variations in relative composition may also be reflected in simple weight ratio comparisons. The following examples are reflected in weight percentages and reflect the number of grams used to prepare the compositions.

[0019] When water is added to form the film-forming polymeric composition, the units in the following examples are expressed in either weight percentages or in milliliters (mls). It is understood, however, that any quantities within the claimed ratios may be prepared depending upon the needs of the preparer and upon the quantity needed to manufacture.

EXAMPLE 1

[0020]

	wt%
Low-acyl gellan gum	19.05%
k-carrageenan	47.62%
locust bean gum	23.81%
Potassium citrate	9.52%

EXAMPLE 2

[0021]

High-acyl gellan gum	19.05%
k-carrageenan	47.62%
locust bean gum	23.81%
Potassium citrate	9.52%

The gum blends such as those described in Examples 1 or 2 or other gum blends within the scope of the present invention may be further combined with additional ingredients to form a film-forming polymer composition. The following examples are representative of those film-forming polymeric compositions useful for the production of capsules and the like and are not to be construed as limiting the scope of the present invention.

EXAMPLE 3

[0022]

Gum blend (EX 1)	4.00 %

(continued)

Sorbitol	5.67 %
Glycerine	5.67 %
Corn syrup	5.67 %
Deionized water	78.99 %

EXAMPLE 4

[0023]

Gum blend (EX 2)	4.00 %
Sorbitol	5.67 %
Glycerine	5.67 %
Corn syrup	5.67 %
Deionized water	78.99 %

In addition to the examples described above, the following film-forming polymeric compositions may also be prepared.

EXAMPLE 5 (C)

[0024]

Gellan gum (LA)	0.66 %
k-carrageenan	0.66 %
locust bean gum	0.66 %
Glycerine	0.60 %
sodium citrate	0.10 %
Potassium chloride	0.10 %
Deionized water	97.22 %

EXAMPLE 6 (C)

[0025]

Gellan gum (HA)	0.40 %
k-carrageenan	1.00 %
Locust bean gum	1.00 %
Polydextrose	13.40 %
Sorbitol	4.00 %
Sodium citrate	0.10 %
Potassium chloride	0.10 %
Deionized water	80.0 %

EXAMPLE 7 (C)

[0026]

Gellan gum (HA)	0.29 %
k-carrageenan	1.62 %
locust bean gum	0.81 %
Sorbitol	6.00 %
Glycerine	6.00 %
corn syrup	6.00 %

(continued)

Potassium citrate	0.29 %
Deionized water	78.99 %

EXAMPLE 8 (C).

[0027]

Gellan gum (LA)	0.66%
k-carrageenan	1.33%
konjac	2.33%
Glycerine	9.97%
Sodium citrate	0.30%
Deionized water	85.41%

[0028] The physical properties of examples 3-7 are listed below and demonstrate the effectiveness of the films prepared within the scope of the present invention. In addition to the described physical properties, the capsules or other products formed from the film-forming compositions are particularly useful because they provide a gelatin-free polymeric composition.

Example Number	Tensile Strength (psi)	Elongation at break (%)
3	525	143
4	531	177
5	2423	78
6	1087	107
7	396	122

Films of the example solutions while hot were cast with a knife blade set at 100 mil clearance and allowed to air dry 24 hours at 60 % relative humidity and 22 °C prior to being tested on an INSTRON Model 1011 tensile-testing instrument.

EXAMPLE 9 (C)

[0029] The composition was prepared according to the following process:

(1) A tri-gum blend comprising gellan gum, locust bean gum and k-carrageenan gum in the relative ratios as described above such as, for example, (a) high-acyl gellan gum (.29 grams); (b) k-carrageenan gum (1.62 grams); (c) locust bean gum (.81 grams); and a salt such as potassium citrate (.29 grams) wherein the potassium citrate is used as a sequestrant/gelling salt was mixed into cold (20-30°C) deionized water (about 80 mls) and then the ingredients sorbitol (6 grams), glycerine (6 grams) and corn syrup (6 grams) were added with agitation. The mixture was then heated to a temperature of about 90 °C with agitation and held at this temperature for about 10 minutes; (2) The hot solution produced in step 1 was then, for example, transferred to an encapsulation machine well known to those skilled in the art which was first developed in 1932 by R.P. Scherer, wherein a 0.76 mm - 1.02 mm (30-40 mil) film was formed on rotating steel drums. The rotating steel drums can vary in size from 30 cm (12 inches) in diameter to 75 cm (30 inches) or more depending on the size of the encapsulation machine. The width of the rotating steel cylinder can also vary from 15-30 cm (6-12 inches). The drums are hollow and can be heated or cooled by injecting liquids within the drum. The two films thus formed on the steel drums proceed through counter rotating dies that were designed to simultaneously form, cut and fill various sized and shaped capsules. The counter-rotating dies are normally made of solid brass and are cylinders approximately six inches in diameter and 30 cm (12 inches) long depending on the width of the encapsulating film. Various shapes of capsules may be cut into the brass circumfrence of the die. The fill material may be either solid or liquid material.

[0030] The fill material used in the above examples was mineral oil and fragrances since this material may be utilized in the production of bath beads which generally contain mineral oil and fragrances. Of course, the fill-material may be

selected from any desired known solid or liquid material which the manufacturer deems necessary. The edges of the capsule formed in this process were heat and pressured sealed at a temperature range of 75-85°C and a pressure range of 0.1-2 psi and the filled capsules were then washed with a petroleum based solvent to remove the traces of mineral oil, dried to a pre-determined moisture content of 3-4% and packaged. The moisture content of the film during the encapsulation process controls the melting temperature and the proper sealing of the capsules.

[0031] Capsules produced in the above process can further be utilized in a method of treating a nutritional deficiency in a mammalian organism in need of treatment thereof comprising administering to said mammalian organism an edible soft capsule of the composition according to claim 1, wherein the capsule contains contents selected from an essential vitamin or nutrient and a method of administering a bath capsule to a bath comprising adding to said bath a soft capsule of a water soluble composition according to claim 1, wherein the capsule contains contents selected from a bath oil or detergent or perfume. Of course, additional ingredients may be added to the capsule contents during the manufacturing process to produce a suitable target capsule.

[0032] Gelatin-free carbonless paper utilizing microcapsules containing dyes may readily be prepared using the claimed composition and process. Furthermore, the present invention also encompasses microencapsulation of any water-immiscible liquids to, for example, mask unpleasant tastes or to protect substances from oxidation and also to allow controlled release of encapsulated material and physical separation of reactive materials.

Claims

1. A tri-gum blend comprising (a) low -acyl gellan gum with a relative weight percentage of about 20% ; (b) k-carrageenan gum with a relative weight percentage of about 50%; (c) locust bean gum, or konjak gum or mixtures of locust bean gums and konjak gums in a ratio of 1:100 to 100:1, with a relative weight percentage of about 25%; and potassium citrate in a relative weight percentage of about 9%.

2. A gelatin-free soft capsule wherein the capsule contents are selected from a solid or liquid and comprising from 1 to 10 weight percent of the total weight of the capsule of a tri-gum-blend composition of Claim 1.

3. The capsule according to Claim 2, comprising a plasticizer selected from sorbitol, glycerine, propylene glycol, polyethylene glycol, corn syrup, sucrose, fructose and combinations thereof.

4. A process for producing a gelatin-free soft capsule according to any of Claims 2-3, comprising the steps of:

   (a) transferring a hot aqueous mixtures containing the gum blend as defined in Claim 1 to a plurality of rotating drums;

   (b) casting a 0.76 mm - 1.02 mm [30-40 mil] films over rotating drums;

   (c) passing at least two of the 0.76 mm - 1.02 mm [30-40 mil] films over rotating dies;

   (d) simultaneously forming, filling with contents selected from a liquid or a solid, heat-sealing and cutting a plurality of capsules; and

   (e) washing and drying said capsules

5. The process according to claim 4, wherein the capsule contains contents s elected from bath oil, detergent, perfume, essential vitamins or nutrient.

Patentansprüche

1. Drei-Gummi-Mischung, die (a) Niederacylgellangummi mit einem relativen Gewichtsprozentsatz von ungefähr 20 %; (b) κ-Carragenangummi mit einem relativen Gewichtsprozentsatz von ungefähr 50 %; (c) Robinienbohnengummi oder Konjakgummi oder Mischungen aus Robinienbohnengummis und Konjakgummis in einem Verhältnis von 1:100 bis 100:1 mit einem relativen Gewichtsprozentsatz von ungefähr 25 %; und Kaliumcitrat in einem relativen Gewichtsprozentsatz von ungefähr 9 % umfaßt.

2. Gelatinefreie Weichkapsel, deren Kapselinhaltsstoffe flüssig oder fest sind und die 1 bis 10 Gew.-%, auf Basis des

Gesamtgewichts der Kapsel, einer Drei-Gummi-Mischungszusammensetzung gemäß Anspruch 1 umfaßt.

3. Kapsel gemäß Anspruch 2, die einen Weichmacher umfaßt, ausgewählt aus Sorbitol, Glycerin, Propylenglycol, Polyethylenglycol, Maissirup, Sucrose, Fructose und Kombinationen daraus.

4. Verfahren zur Herstellung einer Gelatine-freien Weichkapsel gemäß mindestens einem der Ansprüche 2 bis 3, das folgende Schritte umfaßt:

(a) Übertragung einer heißen, wäßrigen Mischung, die die Gummimischung gemäß Anspruch 1 enthält, in eine Mehrzahl an Drehtrommeln,

(b) Gießen eines 0,76-1,02 mm (30-40 mil) Films über die Drehtrommeln;

(c) Passieren von mindestens zwei der 0,76-1,02 mm (30-40 mil) Filme über rotierende Stempel;

(d) gleichzeitiges Formen, Befüllen mit aus einer Flüssigkeit oder einem Feststoff ausgewählten Inhaltsstoffen, Warmverschweißen und Schneiden einer Mehrzahl von Kapseln, und

(e) Waschen und Trocknen der Kapseln.

5. Verfahren gemäß Anspruch 4, worin die Kapsel Inhaltsstoffe enthält, die ausgewählt sind aus Badeöl, Reinigungsmittel, Parfum, essentiellen Vitaminen oder Nährstoffen.

Revendications

1. Mélange de trois gommes comprenant (a) une gomme gellan faiblement acyle avec un pourcentage en masse relatif d'environ 20% ; (b) une gomme k-carraghénine avec un pourcentage en masse relatif d'environ 50% ; une gomme de caroube ou une gomme de konjak ou des mélanges de gommes de caroube et de gommes de konjak dans un rapport compris entre 1:100 et 100:1, avec un pourcentage en masse relatif d'environ 25% ; et du citrate de potassium avec un pourcentage en masse relatif d'environ 9%.

2. Capsule molle sans gélatine où les contenus de la capsule sont choisis à partir d'un solide ou d'un liquide, comprenant de 1 à 10 pour cent en masse du poids total de la capsule d'un mélange de trois gommes selon la Revendication 1.

3. Capsule selon la Revendication 2, comprenant un plastifiant choisi parmi le sorbitol, la glycérine, le propylène glycol, le polyéthylène glycol, le sirop de glucose, le saccharose, le fructose et des mélanges desdits produits.

4. Procédé de production d'une capsule molle sans gélatine selon une quelconque des Revendications 2 à 3, comprenant les étapes suivantes :

(a) transfert d'un mélange aqueux chaud contenant le mélange de gommes défini dans la Revendication 1 vers une pluralité de tambours rotatifs ;

(b) coulée de films de 0,76 mm à 1,02 mm sur les tambours rotatifs ;

(c) passage d'au moins deux des films de 0,76 mm à 1,02 mm sur des matrices rotatives ;

(d) simultanément, formage, remplissage avec des contenus liquides ou solides, soudage à chaud et découpe d'une pluralité de capsules ; et

(e) lavage et séchage desdites capsules.

5. Procédé selon la Revendication 4, où le contenu de la capsule est choisi entre une huile de bain, un détergent, un parfum, des vitamines essentielles ou un nutriment.